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IS 12135 (1987): Method for determination of acetic acid content of acetate or triacetate fibres [TXD 5: Chemical Methods of Test]



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Indian Standard

METHOD FOR DETERMINATION OF
ACETIC ACID CONTENT OF ACETATE
OR TRIACETATE FIBRE MATERIALS

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHOD FOR DETERMINATION OF ACETIC ACID CONTENT OF ACETATE OR TRIACETATE FIBRE MATERIALS

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Indian Standard

METHOD FOR DETERMINATION OF ACETIC ACID CONTENT OF ACETATE OR TRIACETATE FIBRE MATERIALS

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 31 August 1987, after the draft finalized by the Chemical Methods of Test Sectional Committee had been approved by the Textile Division Council.

0.2 Acetic acid content of acetate or triacetate fibres is an important parameter for controlling the degree of acetylation of regenerated cellulose and is useful in ascertaining chemical damage to acetate or triacetate fibres during processing by cuprammonium fluidity test.

0.3 In reporting the result of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes a method for determination of acetic acid content of acetate or triacetate fibre materials.

1.2 The method prescribed in this standard is not applicable to acetate and triacetate fibre materials containing sizing and finishing substances such as polyvinyl acetate, polyacrylic or polymethacrylic acid, cellulose ether, carbonic acid, etc, the presence of which may falsify the results of titration.

1.3 While testing dyed or printed specimens, there is a possibility that the dye may get dissolved and hence make it difficult to recognize the colour change during titration. In such a case, the titration should be carried out electrometrically.

*Rules for rounding off numerical values (revised).

2. PRINCIPLE

2.1 The acetate fibres are saponified with alcoholic potash lye. The acetic acid content is determined by the amount of alkali used to reach the end point, titrimetrically.

3. SAMPLING

3.1 Lot — The quantity of same type and quality of acetate or triacetate fibre material and of the same form (fibre, yarn or fabric, etc) delivered to a buyer against one despatch note shall constitute a lot.

3.1.1 If the textile material is fibre or yarn and the lot consists of more than 200 kg of fibre or yarn, it shall be divided into sub-lots each weighing 200 kg or less.

3.1.2 Each sub-lot shall be tested separately.

3.2 Sampling for Fibre and Yarn

3.2.1 From a sub-lot, 15 increments, each weighing approximately 10 g, shall be taken from different parts and mixed thoroughly. This shall constitute a test sample.

3.3 Sampling for Fabrics

3.3.1 Unless otherwise specified, the number of pieces to be selected from a lot shall be in accordance with Table 1. The pieces thus selected shall constitute a gross sample.

TABLE 1 SAMPLE SIZE

LOT SIZE (PIECES)	SAMPLE SIZE (PIECES)
(1)	(2)
Up to 100	3
101 „ 300	4
301 „ 500	5
501 and above	7

3.3.2 From each piece in the gross sample selected as in **3.3.1**, cut out small portions from at least two different parts weighing about 25 g. The parts selected shall be as representative as possible of the gross sample. In the case of fabrics with a definite repetition in weave pattern, the parts selected shall include all yarns in the complete pattern. Dissect small portions of the fabric thus collected into yarns and mix them thoroughly. This shall constitute a test sample.

3.4 If the test sample consists of yarn, woven material or hosiery or knitted fabric, they shall be separated into yarn pieces of 1 to 2 cm length and mixed thoroughly to prepare the test sample.

4. APPARATUS

4.1 Weighing Glass

4.2 Desiccator with Blue Gel Filling

4.3 Analytical Balance with an Accuracy up to 1 mg

4.4 Air Drying Oven Capable of Maintaining at $105 \pm 2^\circ\text{C}$

4.5 1 000 ml Measuring Flask

4.6 200 ml Erlenmeyer Flask with Ground Stopper

4.7 50 ml Pipette

4.8 Burette

4.9 1 000 ml Erlenmeyer Flask

5. REAGENTS

5.0 Quality of Reagents — Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS : 1070-1977*) shall be used.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the test results.

5.1 Alcoholic Potash Lye — 1 M, potassium hydroxide (free of carbonate) in tablet form dissolved in ethanol.

5.2 Hydrochloric Acid — 1 M.

5.3 Caustic Soda Lye — 1 M.

5.4 Phenolphthalein Solution — 1 g phenolphthalein dissolved in 95 ml of ethanol and 5 ml distilled water.

6. PREPARATION OF TEST SPECIMEN

6.1 From the test sample (*see* 3.2, 3.3 and 3.4) after removing size and finishing as recommended in IS : 9068-1979†, draw a representative specimen weighing 2.0 ± 0.1 g. Cut test specimen into pieces of approximately 25 mm length. Take at least two such test specimens.

*Specification for water for general laboratory use (*second revision*).

†Recommended methods for the removal of non-fabrous matter prior to quantitative analysis of fibre mixtures.

7. PROCEDURE

7.1 Dry the test specimen (**6.1**) in the weighing glass at $105 \pm 2^{\circ}\text{C}$ temperature in a drying oven to constant mass. The mass shall be taken as constant when the difference between two successive weighings at an interval of 20 minutes does not exceed 0.1 percent. Cool the dried test specimen in the desiccator and weigh it correct to 1 mg.

7.2 Transfer the weighed test specimen (**7.1**) into a 200 ml Erlenmeyer flask. Add to it 50.0 ml of 1 M alcoholic potash lye, close the flask and leave it for 48 hours at room temperature. Then add to it 50.0 ml of 1 M hydrochloric acid, shake the flask thoroughly for 5 minutes and leave it for one more hour at room temperature.

7.3 Wash the contents of the Erlenmeyer flask with about 500 ml of distilled water in the 1 000 ml Erlenmeyer flask.

7.4 Titrate the contents of the flask (**7.3**), after addition of three drops of phenolphathelein solution with 1 M of caustic soda lye.

7.5 Find out the amount of caustic soda lye used in ml.

7.6 Repeat the procedure from **7.1** to **7.5** with other test specimens and find out the amount of caustic soda lye consumed, in ml, for each test specimen.

8. CALCULATION

8.1 Calculate the acetic acid content of all the test specimens separately with reference to dried fibre material by the formula:

$$\text{Acetic acid content, percent} = \frac{6 \times a}{E} \times 100$$

where

a = the amount of 1 M caustic soda lye in ml (see **7.5** and **7.6**),
and

E = the weighed amount of the dried fibre material in g
obtained in **7.1**.

8.2 Find out the average acetic acid content, percent of the fibre material.

9. REPORT

9.1 The test report shall indicate the following:

- a) Type and quality of the textile material tested, and
- b) Acetic acid content, percent, rounded to three significant figures (individual values and the mean value separate).

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INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

<i>Quantity</i>	<i>Unit</i>	<i>Symbol</i>	<i>Definition</i>
Force	newton	N	$1 \text{ N} = 1 \text{ kg.m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s (s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volts	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$